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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

YUUKI TAUCHI, ET AL. : EXAMINER: MORILLO, J.C.

SERIAL NO: 10/633,550 :

FILED: AUGUST 5, 2003 : GROUP ART UNIT: 1742

FOR: AG BASE ALLOY THIN FILM AND : SPUTTERING TARGET FOR FORMING

AG BASE ALLOY THIN FILM

DECLARATION UNDER 37 C.F.R. §1.132

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Junichi Nakai who deposes and states that:

- 1. I have a Ph.D. in engineering (electronics engineering), which was conferred upon me in March, 1997 by Tohoku University located in Sendai prefecture.
- 2. I have been employed by Kobe Steel, Ltd. since 1989 and I have a total of 18 years of work and research experience in the field of thin film materials for magnetic and optical thin films.
- 3. I understand the English language or, at least, the contents of this Declaration were made clear to me prior to executing the same.
- 4. The following experiments were carried out by me or under my direct supervision and control.
 - 5. The following is a description of the experiments performed:

Using alloy sputtering target, a thin film of pure Ag or Ag-Bi alloys (samples No. 1-8 of Table 1) was deposited on a substrate shown below with a DC magnetron sputtering process using a Unaxis Cube-star sputtering equipment, under the following conditions:

Composition of target: Ag-0.1at%Bi for sample No.1-7 and Pure Ag for sample No.8

DC cathode: ARQ930

Sputtering power: 2kW

Substrate: Corning #1737 glass (1cm square) for samples of measurement of HRBS, or Polycarbonate of specular surface (0.6mm thickness, $12 \text{cm} \phi$) for samples of measurement of ICP and Optical properties

The Bi content was adjusted depending on deposition conditions described in Table 1. The Bi content of each sample was measured by Inductively Coupled Plasma (ICP) Atomic Emission Spectroscopy and High-resolution Rutherford backscattering Spectrometry (HRBS). Bi₂O₃ film thickness and Bi concentration gradient of each sample was measured by HRBS. The method of measurement of optical properties is described below.

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Table 1

| No. | Deposition condition | | Film | Bi content | | Bi2O3 film thickness | Bi concentration gradient | Optical properties | |
|-----|----------------------|-----------------------|-------------------|--------------|---------------|----------------------------|---------------------------------|--------------------|---------------------------|
| | Ar flow rate (sccm) | Deposition period (s) | thickness (nm) | ICP (at%) | HRBS* (at%) | HRBS* (Å) | HRBS | Reflectivity (%) | Transmit- tance (%) |
| 1 | 13 | 2 | 87 | 0.41 | 0.5 ± 0.1 | 2.0 ± 0.1 | No significant gradient | 88.6 | 0.8 |
| 2 | 20 | 2 | 92 | 0.47 | 0.5 ± 0.1 | 2.2±0.1 | | 86.8 | 0.5 |
| 3 | 25 | 2 | 93 | 0.53 | 0.6±0.1 | 2.2 ± 0.1 | | 85.0 | 0.5 |
| 4 | 30 | 2 | 93 | 0.55 | 0.7 ± 0.1 | 2.3 ± 0.1 | | 83.4 | 0.4 |
| 5 | 70 | 2 | 96 | 0.84 | 0.9±0.1 | 2.1 ± 0.1 | | 74.5 | 0.3 |
| 6 | 30 | 1 | 47 | 0.67 | 0.6±0.1 | 2.3±0.1 | | 76.0 | 11.1 |
| 7 | 30 | 3 | 144 | 0.56 | 0.7±0.1 | 2.3 ± 0.1 | | 82.3 | 0.01 |
| 8 | 13 | 2 | 81 | | - | - | - | 93.3 | 0.6 |

*High-resolution RBS

As can be seen from Table 1, the thicknesses of Bi_2O_3 films are substantially constant regardless of the varying Bi contents. This indicates that the films can have barrier capability even at low Bi contents. It is also apparent that the reflectivity decreases with an increasing Bi content. This constitutes grounds for the specified upper limit of the Bi content.

Among obtained data of Table 1, correlation between Bi content (with ICP) and reflectivity (405 nm) were compared.

Table 2

| Bi content | Bi2O3 film thickness | Optical properties | |
|------------|----------------------|--------------------|--|
| ICP (at%) | HRBS(Å) | Reflectivity (%) | |
| 0.41 | 2.0 ± 0.1 | 88.6 | |
| 0.47 | 2.2±0.1 | 86.8 | |
| 0.53 | 2.2±0.1 | 85.0 | |
| 0.55 | 2.3±0.1 | 83.4 | |
| 0.84 | 2.1 ± 0.1 | 74.5 | |
| 0.56 | 2.3±0.1 | 82.3 | |
| 0.07 | 1.0±0.1 | 92.0 | |
| 0.0 | 0 | 93.3 | |

Data on Ag-0.07Bi obtained in past experiments, which was deposited on Corning #1737 glass substrate with a DC magnetron sputtering process using SHIMADZU HSM-552 sputtering equipment, under the following conditions, were added.

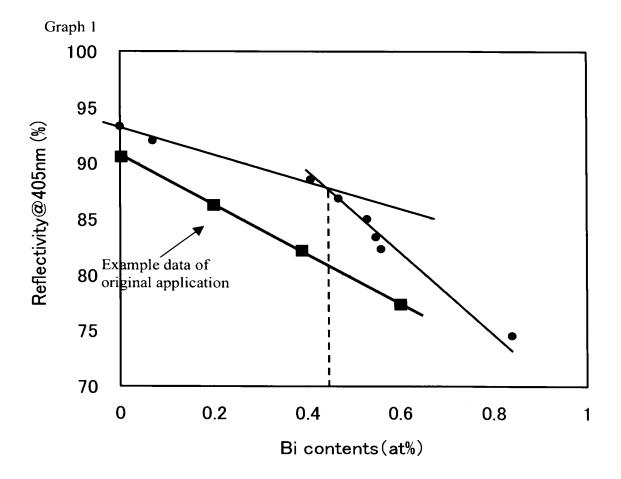
Target: Ag-1.5at%Bi (5mm thickness, 4 inch ϕ)

Sputtering power: DC 200W

Ar pressure: 2mTorr

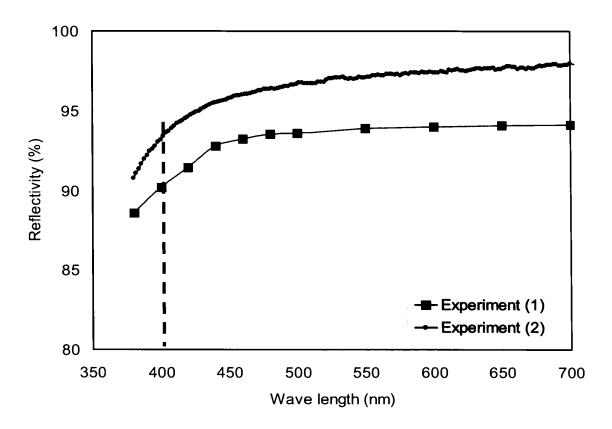
The sample with Bi content of 0.67 atomic percent should be excluded because of its small thickness.

Graph 1 as below shows correlation between Bi content (with ICP) and reflectivity (405 nm).



As illustrated in Graph 1,

- (1) The barrier layers have substantially constant thicknesses at Bi contents of 0.4 atomic percent or more. This indicates that the barrier layers show saturated durability at the specific Bi content or higher.
- (2) The reflectivity decreases with an increasing Bi content. It gradually decreases at Bi contents up to 0.4 atomic percent but sharply decreases at Bi contents of 0.4 atomic percent or more.
- 6. The data of reflectivity illustrated above are slightly different from the data of originally filed. The reason why such inconsistency has occurred is explained below.



Graph 2 Comparison in Spectral Reflectivity of pure Ag

Ex. (1): Measuring device used in Examples of the filed application Ex. (2): Measuring device used in the additional experiments.

From Graph 2 it is apparent that the measured reflectivities at wavelengths around 400 nm vary depending on the precision of measuring devices, because the reflectivity significantly varies with a large gradient depending on the wavelength in this region.

Difference between the measuring device for obtaining the present data and the data in originally filed application is compared in Table 2 below.

Table 2

| | Examples in the filed application | Additional experiments |
|------------------|--|--|
| Measuring device | Polar Kerr Scope NEO ARK | V-570D from JASCO Corporation |
| | MODEL BH-810 from NEOARK | |
| | Corporation | |
| Configuration of | · Relative reflectivities are measured | Absolute reflectivities* are measured. |
| device and other | (calibrated with Al standard mirror) | |
| conditions | • The inclination of sample is | There is no need of correcting |
| | corrected and the wavelength is | inclination of sample. Sweep of |
| | adjusted both manually. | wavelengths and measurements are |
| | | conducted fully automatically. |
| Wavelength | 20 nm per step (the wavelength is | less than 1 nm per step (automatic |
| resolution | adjusted manually on the dial) | sweep) |
| Remarks | A detection unit alone of a device | |
| | which is originally designed for | |
| | another purpose, i.e., the | |
| | measurement of Kerr rotation angle is | |
| | used herein for the measurement of | |
| | reflectivity, and most parameters in | |
| | measurement of reflectivity must | |
| | therefore be controlled manually. For | |
| | measuring reflectivity, a value at | |
| | which the signal of reflected light | |
| | reaches maximum is read while the | |
| | inclination of sample stage is | |
| | adjusted. Thus, the measured | |
| | reflectivity includes a large error. | |
| | (Error bar becomes large) | |

^{*} Absolute reflectivity: When monochromatic light is applied to a sample thin film and light reflected from the thin film is measured, all the reflected light does not always return to the detector, because the reflected light scatters due typically to unevenness of the sample surface and the inclination of the sample. In the measuring device used in Examples of the filed application, calibration is conducted while estimating the scattered light using a standard reference sample, but it is difficult to measure reflectivities precisely, because the degree of scattering varies from sample to sample as set in the measuring device. In contrast, the measuring device used in the additional experiments can carry out measurement of absolute reflectivities without the need of a standard reference sample, because measurement is conducted after concentrating the scattered light.

Application No. 10/633,550 Declaration under 37 C.F.R. §1.132

7. I declare further that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

8. Further Declarant saith not

Name: Junichi Nakai Kobe Steel, Ltd.

Fieb. 5, 2008

Date